

AD-A242 479



2  
JUN  
S DTIC  
ELECTRO-  
ROVE REGD  
C D

QUARTERLY REPORT

N00014-88-C-0681

DEVELOPMENT OF ELEMENTS OF A HIGH Tc  
SUPERCONDUCTING CABLE

Dr. Kenneth W. Lay  
518-387-6147  
FAX 518-387-7495

GE CORPORATE RESEARCH & DEVELOPMENT  
PO BOX 8  
Schenectady, NY 12301

July 1, 1991 - September 30, 1991

Approved for public release;  
Distribution Unlimited

91-14675



91 10 31 044

## **PROGRAM SUMMARY**

The program is aimed at the development of long lengths of silver-clad BSCCO. The material of choice is BSCCO-2223 with 20K the operating temperature goal. Such a tape conductor ultimately could be used in a coil for a magnet, motor or generator.

The program is designed to tackle several key problems with parallel tasks. A large variety of BSCCO powders are under investigation. Silver tubes are packed with superconductor powder and deformed to form tape conductors. An alternate method of making silver-encapsulated superconducting tape is also being pursued. Variations in the powders and processing parameters are used to optimize the tape Jc.

Progress continues on several fronts as we clarify the conditions required to obtain high Jc tapes. In this quarter:

- 1) An internally oxidized silver magnesium alloy was shown to harden (instead of soften) during 830°C air heat treatments. Plates of this alloy have been prepared and tubing fabrication is underway to make PIT tape.
- 2) Unanticipated increases in Jc were seen for samples heat treated in a 5% O<sub>2</sub> atmosphere at 780°C. This has implications on the best cooling environment for the tapes after the final heat treatment. The reason for the Jc increase is now under study and may explain some of the variations in Jc values seen between different compositions.
- 3) The determination of the quantitative effect of processing parameters on Jc continues. Optimum tape thicknesses are about 0.18mm. Wires configured into a rectangle by turks head rolling before the final tape rolling operations consistently give higher Jc values than tapes made from round wire preforms.

## **PROGRESS ON TASK 1 -- POWDER PRODUCTION**

The goal of this task is the production of reproducible, large powder batches of BSCCO to be used in the tape development effort.

Fully reacted 2223 powders do not result in the best tapes. The highest  $J_c$  values are obtained when the reaction to form 2223 occurs during the final heat-treatment processing of the composite tapes. The initial powder should contain the 2212 phase but with the overall composition designed to readily form the 2223 phase. The ideal powder would be mostly Bi-2212 but should react quickly and completely to Bi-2223. We are pursuing two different approaches. In one the initial powder is a composition known to react readily to 2223 but which is only partly reacted before tape fabrication. In the second method, the initial powder is a mixture of fully reacted 2212 and other phases. In both cases we are considering cation ratios differing from the nominal 2223 values. All powders contain some lead substituted for the bismuth to stabilize the 2223 phase. The addition of excess calcium and copper greatly increases the reaction rate.

We are continuing to explore variations on initial powder compositions.

#### **PROGRESS ON TASK 2 -- TAPE FABRICATION USING POWDER-IN TUBE**

Deformation of BSCCO-filled silver tubes is done by sequential swaging, drawing, and rolling. The tapes are then subjected to final deformation and heat-treatment cycles under task 5 to optimize properties, especially  $J_c$ . The aim of this task is to provide tapes for the  $J_c$  optimization studies.

A process for fabricating tapes as thin as 0.1mm is in-hand. Typically, 0.11m long powder columns packed into 6.35mm diameter silver tubes result in 2.7m long tapes 0.25mm thick and about 3mm wide. Further rolling can reduce the tape thickness to 0.1mm if desired. We have made 14 meter long 0.25 mm thick tapes using commercially available 0.5m silver tubes. Longer lengths can be made depending on the availability of longer and/or larger OD starting tubes.

The initial powder to silver ratio determines the ratio in the final tape. We granulate our initial powders to increase the powder filling factor. Using thin-walled silver tubes we have obtained up to 40 volume % superconductor in the tapes which can be compared with typical reported values of about 25%. Swaging is done to densify the powder column, thicken the silver wall and work harden the silver. Most of the diameter reduction and fiber lengthening is done by drawing.

A more uniform superconductor thickness across the final tape is obtained if the final operation before tape rolling converts the cross section from round to rectangular using a turks head roll set. The diameter at the transition from drawing to rolling controls the final tape width. We typically draw to 1.3 - 1.5mm which gives 2.3 - 2.8 mm wide tapes at 0.25 mm thickness. During the rolling operation the Bi-2212 in the superconductor core is aligned with the basal planes perpendicular to the thickness of the tape.

Since the ultimate test of process variables is the tape  $J_c$ , a large number of tapes have been made. We continue to explore the effect of deformation processing variables on the properties of final tape.

#### PROGRESS ON TASK 3 -- ALTERNATE TAPE FABRICATION PROCESS

An alternate process for the fabrication of high-T<sub>c</sub> conductor Silver-Clad Tape (SCT) is under study. The first step was to have been extrusion of a thin tape containing BSCCO and organic binder. The extruded tape would be partially wrapped in silver foil and heat-treated in an oxidizing atmosphere to remove the organics from the composite tape.

An improvement in the process is feasible. We have demonstrated the ability to fill a silver u-shaped channel with Bi-2223 precursor powder, compress the powder, fold the edges, and diffusion bond the silver-clad package. Such a package has been made and processed by rolling to a thickness of 0.25mm. The superconductor to silver ratio of these composite SCT tapes are in the same range as for PIT processed tapes. This process modification has the advantage over the original concept of not requiring a binder removal step. The length of time required for binder removal from an extruded preform would have made a rapid continuous tape fabrication process difficult.

#### PROGRESS ON TASK 4 -- SILVER FOIL SEALING

The second step in the GE SCT process is the sealing of the edge of the silver foil wrapped around the BSCCO tape.

The silver foil sealing was to be done using pressure welding. An improvement in the process has been demonstrated. If a lap joint of the silver foil is simply pressed together, the silver cladding diffusion bonds during the superconductor heat treatment process. The bond survives subsequent deformation (rolling or pressing) of the tape.

The SCT process now under consideration involves wrapping silver foil around a packed superconductor powder core and heating to seal the silver edges together. The rectangular cross section silver-clad composite can then be processed in a similar manner to as turks head rolled PIT tape preform.

#### PROGRESS ON TASK 5 -- SUPERCONDUCTOR OPTIMIZATION

This key task studies the optimization of the  $J_c$  of silver-clad BSCCO tapes.

Emphasis in this task continues on optimization of heat treatment and deformation conditions to maximize  $J_c$ . Many iterations are required to determine the best set of processing parameters. This involves choice of the correct powder as well as the optimum deformation and heat treatment cycles.

Transport  $J_c$  measured at 77K with no applied field is used as a rapid measure of sample quality. The critical current at 20K in an applied field is approximately given by  $I_c(20K, 2T) = 5 I_c(77K, 0T)$ . More detailed measurements as a function of temperature and field can be done on selected samples.

We have conclusively shown that the best  $J_c$  values in PIT tapes are obtained starting with 2212 powders and reacting to 2223 after fabrication of the tape configuration. The initial powder can either be a partially reacted mix which has not yet been totally reacted to Bi-2223 or it can be a mixture of pre-reacted Bi-2212 and other phases. The Bi-2212 in the tape is aligned during the tape rolling operation.

The final processing of PIT tapes involves a series of heat treatment and deformation cycles. The optimum heat treatment temperatures (in air) for our compositions are in the range 825 - 840°C. Higher temperatures drastically degrade the samples by a partial melt involving the silver sheath. Lower temperatures do not develop the necessary well-sintered 2223 phase. Rolling or pressing operations are needed to densify the superconducting core. We have shown that pressing is much superior to rolling to obtain high  $J_c$  values. We have built a special pressing jig to allow semicontinuous pressing of long tapes.

Attachment for  
Task 5  
Date Tab  
Approved  
Classification

Spec AD-A239766  
Classification  
Controlled by Code  
Avail Loc, or  
Spec. Special

A-1

Silver is the material of choice for the sheath of PIT processed tapes containing bismuth based superconductors. It is very ductile and can be extensively cold worked. It would be desirable, however, if the silver hardness could be increased. After the final tape anneals at temperatures over 800°C any work hardening present in the silver clad is annealed out. The soft silver sheath allows for a more easily damaged tape. In addition, there is a possibility that a harder silver sheath will transmit less tensile strain to the superconductor core during the final pressing or rolling operations. These tensile strains can lead to cracking of the core.

We have shown that the addition of a small amount of magnesium to silver results in an alloy which can be internally oxidized during the final tape anneals and results in a hardening of the silver which persists during the very long high temperature anneals. Figure 1 shows hardness data for the magnesium doped silver alloy in comparison with two silver standards. It can be seen that the alloy as-processed shows similar properties to pure silver. When heated in an oxidizing atmosphere, however, the alloy hardens while the pure silver softens. We are in the process of making magnesium-doped silver tubing which will then be used to make superconductor tape for comparison with tape made from conventional silver tubing.

The experiments on quantifying the parameters which influence  $J_c$  of tape continue. An example is shown in Figure 2. Shown are  $J_c$  values for tapes with 2 different compositions, 3 thicknesses, and processed using either drawing or turks head rolling before the rolling operations. It appears that the best tape thickness is about 0.17mm. This results in high  $J_c$  values and is also thick enough to be handled without damage. It also has been found that turks head rolling to form a rectangular cross section wire before the tape rolling operation consistently gives higher  $J_c$  values compared to rolling a round wire.

We also have discovered that the cooling atmosphere after the final heat treatment is an important source of variability in  $J_c$ . For example, simply heating for 4 hours in a 5%  $O_2$  + 95%  $N_2$  atmosphere improves  $J_c$  values of tapes originally heated for a total of 144 hours in air at 830°C. This improvement was seen for three different compositions with  $J_c$  values for 0.25mm tapes increasing from 4800 to 6000, 4900 to 6000, and 2600 to 7800  $A/cm^2$ . We are now looking for the reason for this improvement. It is suspected that some of the 2223 phase may decompose during cooling. This decomposition would be expected to occur at grain boundaries so that a small amount of decomposition could have a major effect on current transport. The formation of alkaline earth plumbate (lead in

the +4 state) is a likely culprit. If this is the case, cooling in a less oxidizing atmosphere reduces the stability of the plumbate such that it is stable only at lower temperatures. If the plumbate formation temperature is low enough, slower kinetics will allow retention of the metastable 2223 during cooling.

#### **PROGRESS ON TASK 6 -- LONG LENGTH AND SINGLE COIL PROPERTIES**

The properties of long lengths will be studied using coiled tapes. Uniformity along the tape is particularly important.

A necessary condition for the processing of long lengths is the ability to coil the tapes so that the heat-treatments can be done in a batch mode. Tapes with thicknesses as high as 0.25mm total thickness must be bent around radii small enough to allow coils to be placed in small furnaces. In addition the final tapes must have reasonable bend tolerances to allow small coils to be made. We found that 0.25mm thick tapes could be bent around a 3" radius with little degradation in  $I_c$ .

We have done final processing (sequential heat treating and pressing) on 0.22mm thick tapes as long as 1.1m. The heat treatments are done with the tapes coiled on 15cm diameter alumina cylinders. A pressing technique is used where 15.2cm segments of the tape are sequentially pressed with about 1.3cm overlaps between pressings. This semicontinuous pressing operation is amenable to automation. No degradation of  $I_c$  is seen in the overlap regions. We are now perfecting final processing and handling techniques for long tapes.

#### **TALKS AND PAPERS THIS QUARTER**

KW Lay, RH Arendt, JE Tkaczyk, and MF Garbauskas, "Silver-Clad Bi-2223 Processing", Talk given at New York State Institute on Superconductivity Conference, Sept. 26, 19910, Buffalo, NY,

JE Tkaczyk, RH Arendt, HR Hart, KW Lay, and FE Luborsky, "Electric Field Versus Current Density Relations for Bi(2223) Tapes", Talk given at DARPA Meeting in Seattle, WA, Sept. 30, 1991

KW Lay, RH Arendt, JE Tkaczyk, and MF Garbauskas, "Factors Affecting  $J_c$  of Silver Clad Bi-2223 Tapes", Talk given at DARPA Meeting in Seattle, WA, Oct. 1, 1991

## **GOALS FOR NEXT PERIOD**

Evaluate tapes fabricated using magnesium-doped silver cladding.

Quantify the factors influencing  $J_c$  of tapes, especially the effect of cooling in various oxygen pressures.

Fabricate longer tapes and determine their critical currents.

## **FINANCIAL STATUS**

All values are cost plus fixed fee total costs.

TOTAL FUNDING REQUIRED FOR EFFORT \$2,424,530  
01Sept88 through 31Dec91 (40 months)

CURRENT AUTHORIZATION 2,068,530  
01Sept88 through 30Sept91 (37 months)

FUNDING EXPENDED TO-DATE 2,138,440  
01Sept88 through 29Sept91 (37 months)

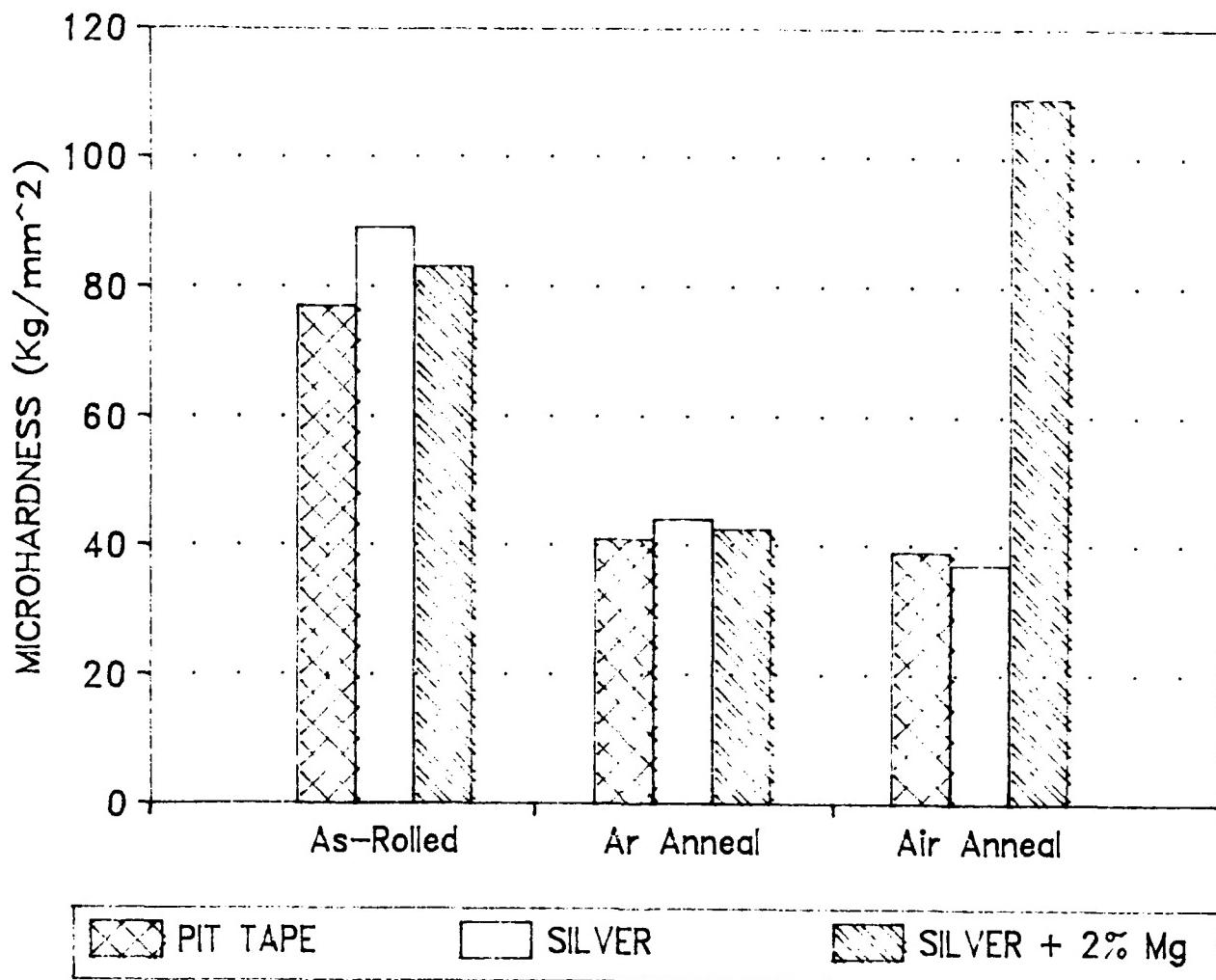


Figure 1 Microhardness of silver cladding on PIT tape, silver tape, and silver + 2 atomic % magnesium tape. Hardness was measured on the surface of tapes as-rolled, after a 30 minute 650°C anneal in argon, or after a 48 hour 830°C anneal in air. The magnesium doped silver is similar to silver during rolling and inert atmosphere annealing so it should be as easily processed into tapes by cold working. It can then be hardened during the final heat treatment of the tapes which are done in oxidizing atmospheres.

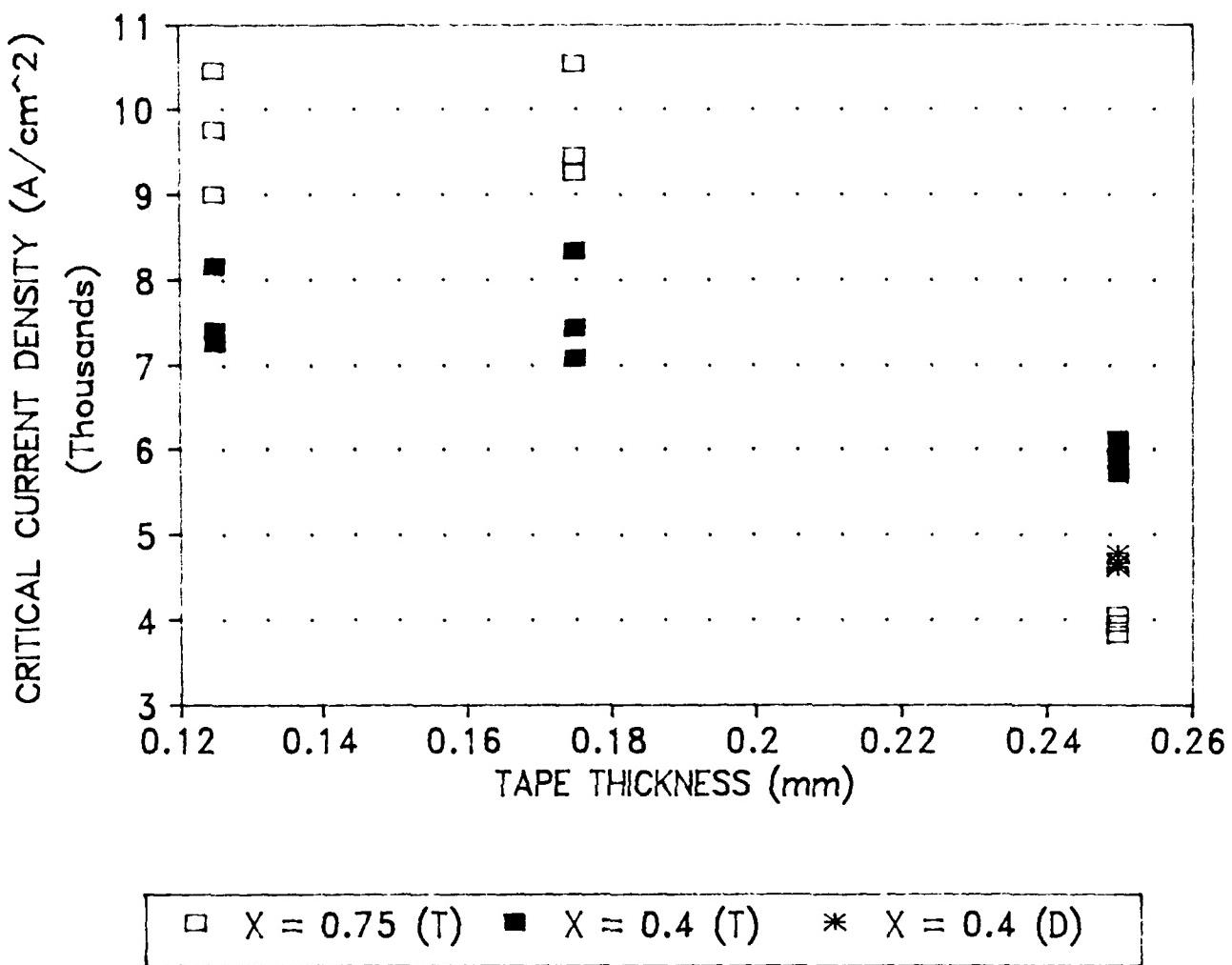


Figure 2 Effect of processing parameters on the  $J_c$  of tapes at 77K in self field. The overall compositions of the tapes are  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{2+X}\text{Cu}_{3+X}\text{O}_Y$  where  $X = 0.4$  or  $0.75$ . The indicated tape thicknesses are as-rolled before the final processing which included 3 heat treatments at  $830^\circ\text{C}$  in air with 2 intermediate pressings at 140,000 psi. One sample set was processed by rolling round wire in the as-drawn condition. The rest were processed by turks head rolling to form a rectangular wire before the tape rolling. Turks head processed tapes about 0.18mm thick are preferred to obtain high  $J_c$  and  $I_c$  values and to be thick enough to minimize handling damage.